New Mexico Tech

June 8, 1992

Dr. James E. Butler
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Naval Research Laboratory
Washington, DC 20375

SUBJECT: JUNE TECHNICAL PROGRESS AND STATEMENT OF FUNDS REPORT
(NRL Contract No. N00014-91-C-2173)

Dear Dr. Butler:

Enclosed is our June technical progress and statement of funds report for the above-referenced contract on "Dynamic Fabrication of Diamond Thermal Management Substrates." We are also including a report prepared by Prof. Dave Goodwin (CalTech) on the thermal conductivity measurements of the shock compacted samples.

Total end-of-May expenditures are: CETR/New Mexico Tech - $ 225,452, CalTech - $ 168,964, and Allied-Signal, Inc., - $ 17,752 (April end).

Yours sincerely,

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JUNE TECHNICAL PROGRESS REPORT

We have conducted careful XRD analysis, which shows that most of the diamond compacts made to-date may contain as much as 5-10% of a non-diamond (possibly graphitic) phase. Electrical resistivity measurements reveals low values, indicating that the diamond particles are perhaps uniformly held together by a skeleton of the non-diamond phase.

We continue to perform shock compaction experiments on various types of pre-treated natural and synthetic diamond powders packed at high initial green densities (> 73% TMD). In the last experiment performed in mid-May, using our 12-capsule plate-impact assembly (at 1.85 km/s velocity), we were able to recover eleven well-consolidated single-piece diamond compacts. The final density of all of these compacts is ≈ 3.3 g/cm^3. We are currently performing XRD analysis and electrical resistivity measurements on these samples, before they are sent for Raman analysis (at Allied-Signal) and thermal conductivity measurements (at CalTech). TEM analysis is also being performed by Prof. Gillian Bond at New Mexico Tech, to identify possible deformation substructures in diamond particle interiors, and possible non-diamond phases at interparticle regions.

A report prepared by Prof. Dave Goodwin (CalTech) on the thermal conductivity measurement technique is enclosed.

The work reported here is done under NRL contract No. N00014-91-C-2173 to CETR, NMT, with subcontracts to CalTech and Allied-Signal, Inc. The COTR at NRL is Dr. Jim Butler (Tel: 202-767-1115), and the Program Manager at DARPA is Dr. William J. Barker (Tel: 703-696-2281).

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Thermal Conductivity Measurement using Pulsed Photothermal Radiometry(PPTR) Technique

We have measured the thermal conductivity of thin samples by Laser Pulsed Photothermal Radiometry (PPTR). Laser Pulsed Photothermal Radiometry is a quick, convenient, non-destructive measurement method. Analyses using a one-dimensional thermal diffusion model are compared with experimental data. Measurements of the thermal conductivity of a molybdenum foil and several shock-consolidated samples have been executed.

Theory

An analytical model describing one-dimensional heat flow was developed to reduce the measured signal to a thermal conductivity for the sample.

For a thin sample (thickness << width), if heat flux is given to the front surface at t=0 as a delta function and there is no heat loss to air at either surface, the temperature rise at the back surface of the sample is given by

$$\frac{\Delta T(t)}{\Delta T(\infty)} = \frac{4}{\sqrt{t^* \pi}} \sum_{n=0}^{\infty} \exp[-(2n+1)^2/t^*]$$

where $t^* = 4\alpha t/L^2$, L is the thickness, α is the thermal diffusivity and t is time. Thermal conductivity k is determined by $k = \rho\alpha c$ where ρ is the density and c is the specific heat.

The non-dimensional temperature rise due to a transient heating pulse was calculated and is shown in Figure 1.

Experimental Technique

Our basic experimental set up is shown in Figure 2. The front surface of the sample was heated by a pulsed Nd:YAG laser beam. Infrared radiation from the back surface of the sample was focused onto a HgCdTe infrared detector. The detector monitored the rise in temperature as heat propagated from the front surface to the back surface. The resistance of the detector is changed proportional to the incident infrared radiation and by this signal we can get the temperature-time history of the back surface. The back surface of the sample was coated with a black paint whose emissivity was assumed to be unity. Noise during excitation pulse was suppressed by using a gated integrator, acquiring one point per laser pulse. The signal-to-noise ratio of the measurement was improved greatly by use of a germanium filter directly over the detector element to exclude scattered laser light.

Experimental Results

Experiments were made for a molybdenum foil and several shock-consolidated diamond samples.

Typical signals obtained from a molybdenum foil are shown in Figure 3. The peak at short times is due to residual stray light. This is followed by a rise due to the increasing infrared radiation detected from the back of the foil. The slight decreasing of the signal above times of 300 μs is due to heat loss to the air. The thickness of this foil was 135 μm. Using this value along with typical handbook values for the density and specific heat for molybdenum at 300 K, thermal conductivity was calculated.

Theoretically, any value of t^* should give the same thermal conductivity. Since experimental results show some deviation from the theoretical temperature rise curve, selecting different values of t^* results in different values of thermal conductivity. For a one-dimensional solution, if we choose $t^*=1$ as a reference non-dimensional time scale, α is given by $L^2/4t$. Since the non-dimensional temperature rise $\Delta T(t)/\Delta T(\infty)$ corresponding to $t^*=1$ is 0.83, we can calculate the α by determining the t which corresponds to this value.

Typical literature values for a molybdenum foil at 300K are $\rho=10240$ kg/m^3, $c=255$ J/kgK and $k=1.39$ W/cmK. The experimental value of k with $t^*=1$ was 1.12 W/cmK. This value is 24% lower than the handbook value. Since errors are mainly generated at the early stage of the signal, we can significantly reduce the error by choosing relatively a high value of t^*. Selecting $\Delta T(t)/\Delta T(\infty)=0.99$, the experimental value of k is 1.31 W/cmK, which is 6% lower than the handbook value. The results for a molybdenum foil are below.

sample	thickness	literature k at 300K	$\Delta T(t)/\Delta T(\infty)$	experimental k	error
moly foil	135 μm	1.39 W/cmK	0.83	1.12 W/cmK	24%
			0.99	1.31 W/cmK	6%

Thermal conductivities for some shock-consolidated diamond samples produced by New Mexico Technology were calculated similarly. The signal obtained from one shock-consolidated diamond sample is shown in Figure 4. For diamond, the handbook values at 300 K are $\rho=3515$ kg/m^3, $c=516$ J/kgK. Thermal conductivity for diamond is dependent on its type and can be as high as 21 W/cmK. The experimental data for shock-consolidated diamond samples are below.

sample#	density(%)	L(mm)	$\Delta T(t)/\Delta T(\infty)$	α(cm^2/s)	k(W/cmK)
9201--02	88.5	0.670	0.83	0.106	0.171
			0.99	0.138	0.220
9201--03	93.3	0.428	0.83	0.096	0.168
			0.99	0.123	0.209
9201--05	92.0	0.575	0.83	0.112	0.187
			0.99	0.169	0.282
9201--07	93.3	0.442	0.83	0.095	0.160
			0.99	0.098	0.166
9201--09	94.0	0.588	0.83	0.099	0.169
			0.99	0.118	0.201

sample#	density(%)	L(mm)	$\Delta T(t)/\Delta T(\infty)$	α(cm^2/s)	k(W/cmK)
9124--04	88.6	0.423	0.83	0.101	0.164
			0.99	0.119	0.191
9131--10*	87.0	1.06	0.83	0.208	0.327
			0.99	0.295	0.465

* fractured sample

The density(%) is the measured density, compared with the theoretical density of diamond, $\rho = 3515\ kg/m^3$. Results show that these shock-consolidated samples have lower than expected thermal conductivities.

The detected signal for one calibrated CVD diamond sample donated by Norton company is shown in Figure 5. The very rapid appearance of the thermal signal on the back surface is evidence of the sample's high thermal conductivity. The rise time is comparable with the detector response time, making it difficult to determine the thermal conductivity of this sample.

Future Work

Several calibrated standard metals and diamond samples should be used to verify the accuracy of the present one-dimensional model.

We should develop an analytical model for a three-dimensional solution accounting for the Gaussian profile heat flux, heat losses from both surfaces, finite absorption coefficients and finite imaged area. Improved methods accounting for the finite detector frequency response should be developed for high thermal conductivity materials.

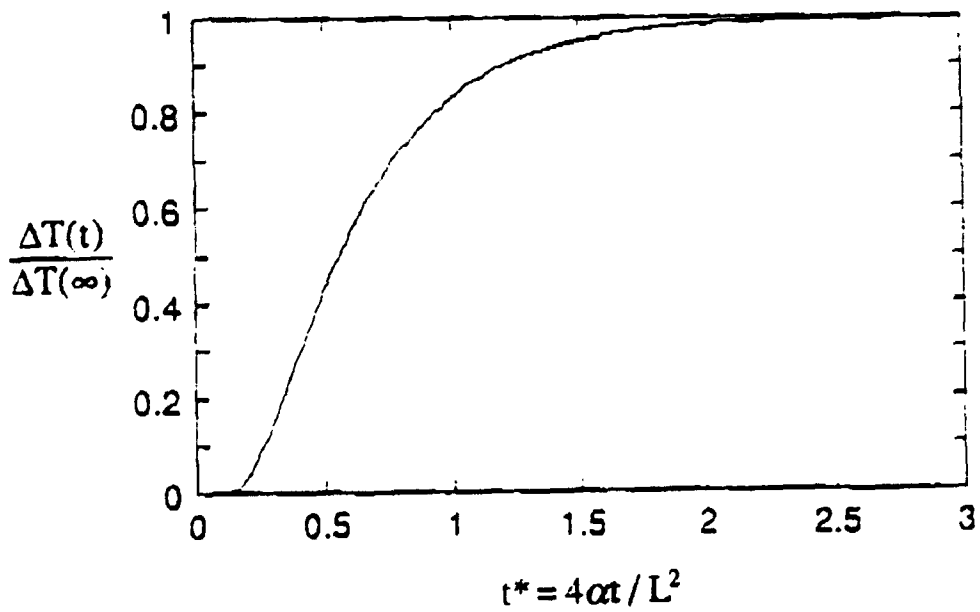

Figure 1. Non-dimensional temperature rise due to transient heating pulse as calculated from one-dimensional theory

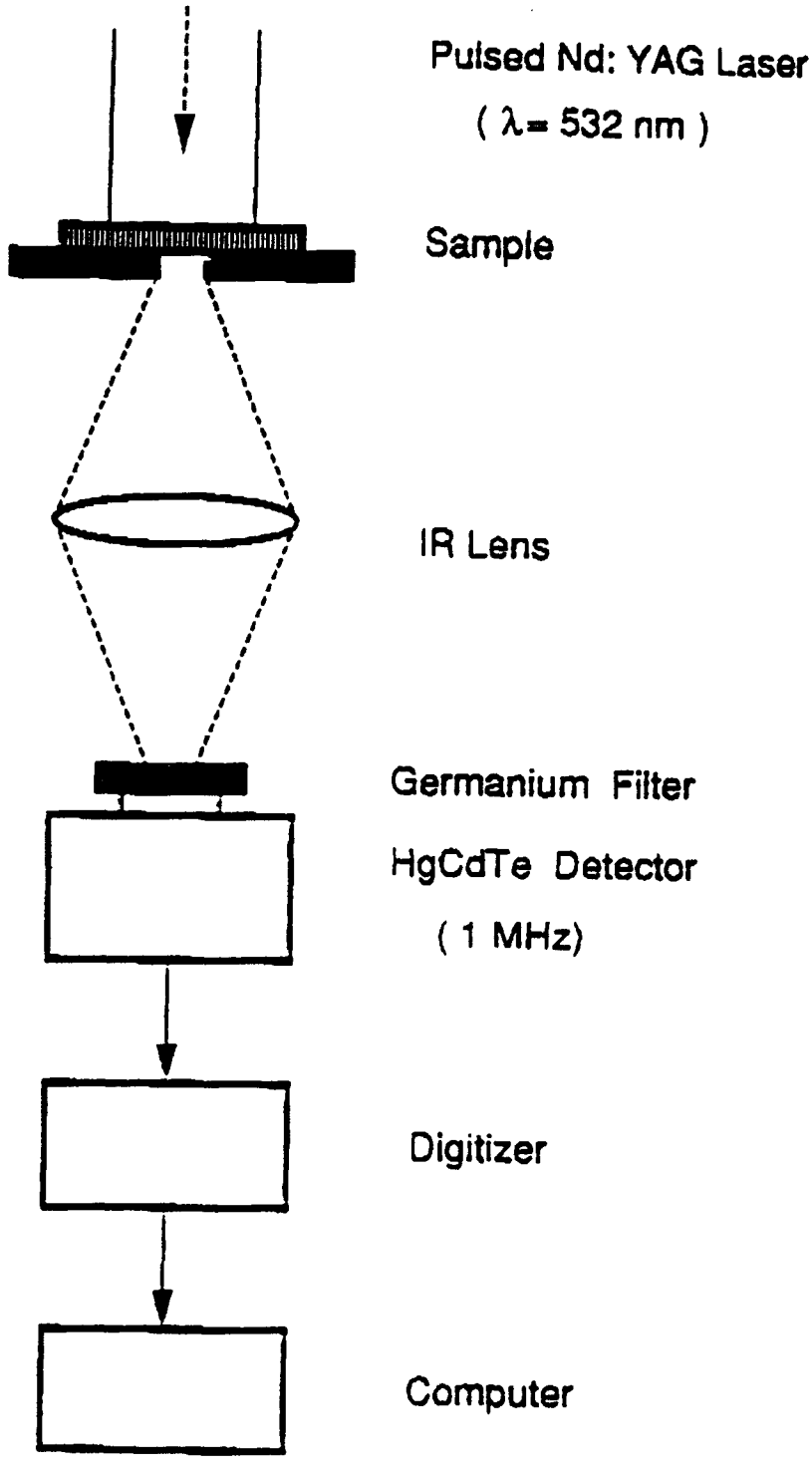

Figure 2. Experimental set up for Thermal Conductivity Measurements

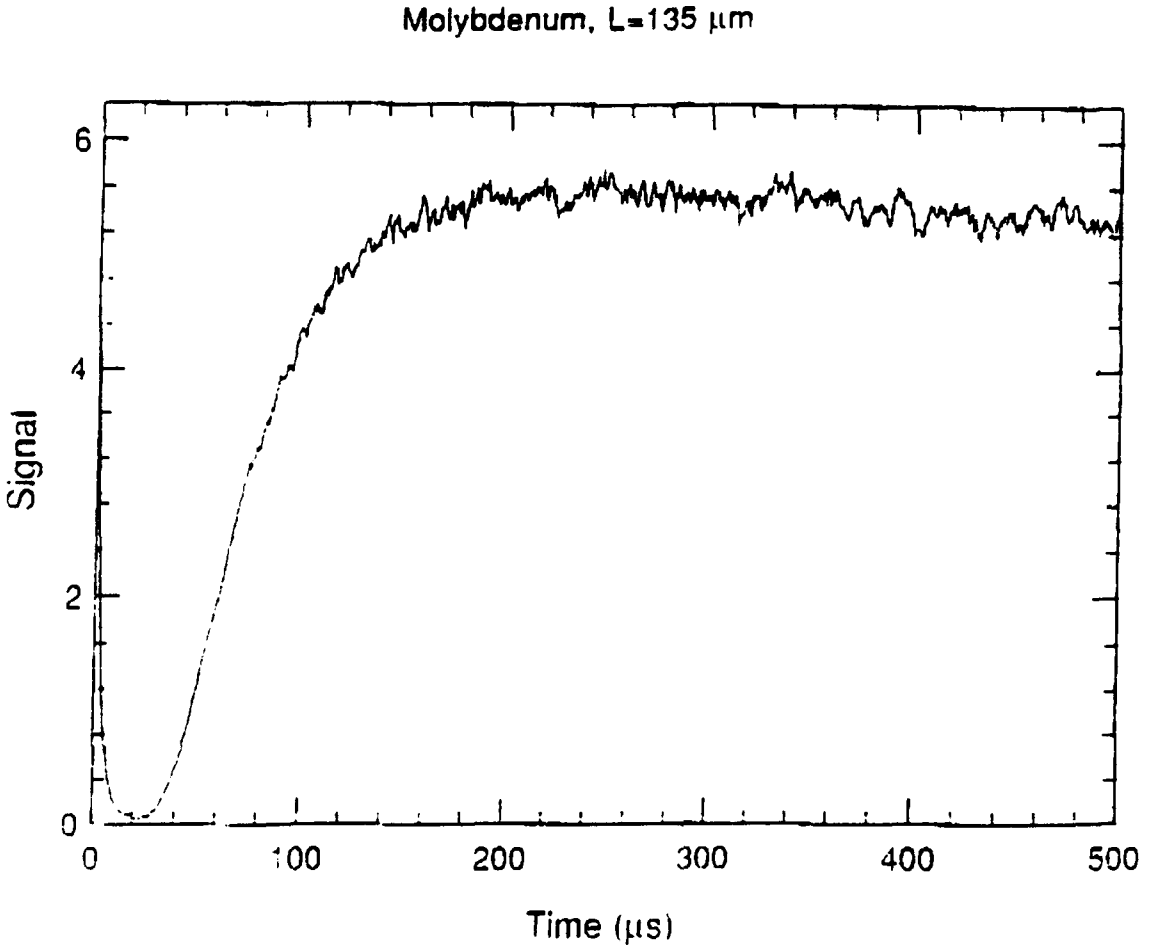

Figure 3. Measured detector signal for a molybdenum foil

9124-08

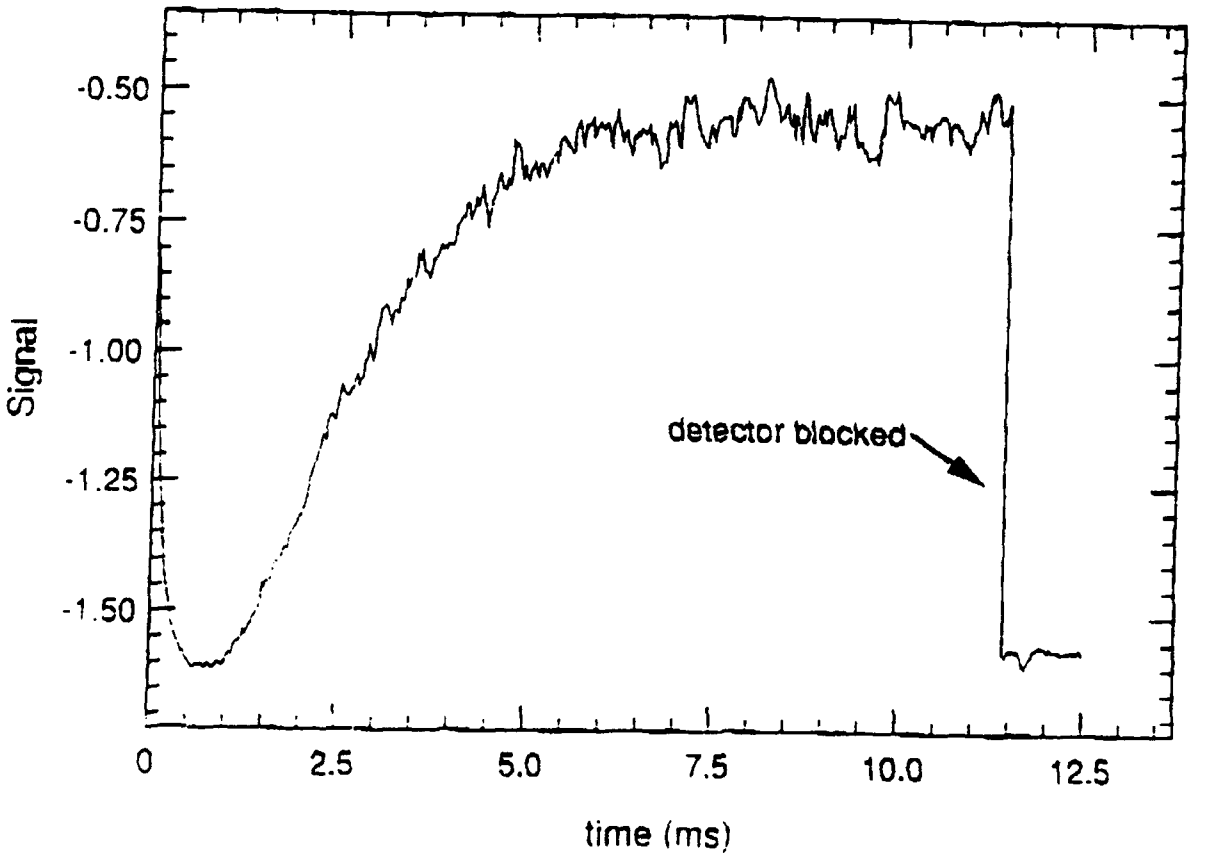

Figure 4. Measured detector signal for a shock-consolidated sample

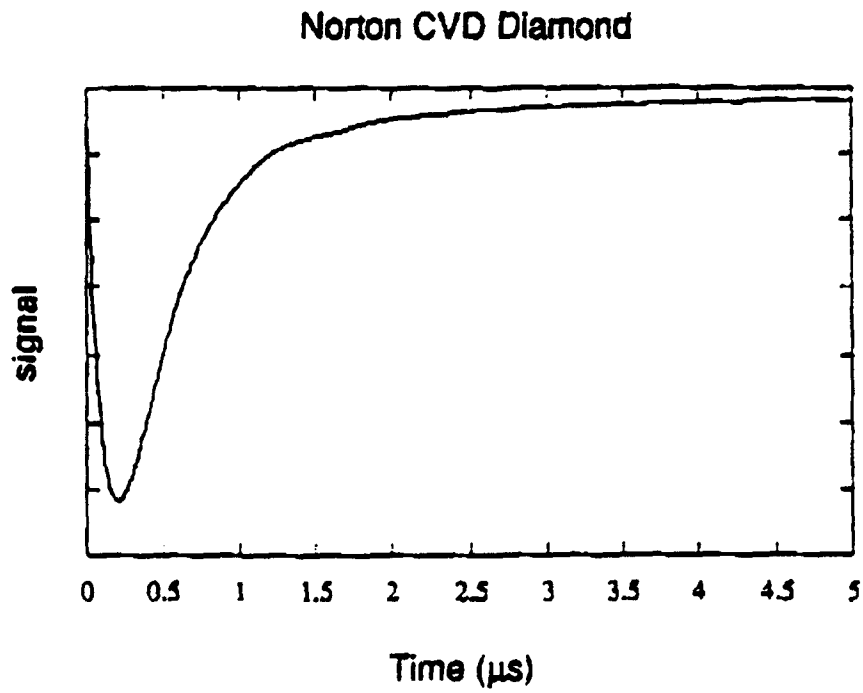

Figure 5. Detector signal from Norton CVD diamond sample TC227